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(71)Applicant : JAPAN ENERGY CORP

(72)Inventor : IWATA YOSHIKI
KATO AKIRA
NAKAJIMA AKIRA

(54) IMPREGNATION OF CATALYST CARRIER

(57)Abstract:

PURPOSE: To provide an inexpensive supporting method of active metal on a catalyst carrier while suppressing the deterioration of the strength of the catalyst carrier at the time of the supporting of active metal by adding an org. solvent wherein the condition of a solubility parameter is specified to a support soln. and specifying the surface tension of the support soln. before impregnating the catalyst carrier.

CONSTITUTION: A support soln. containing one or more kind of active metal selected from Mo, Ni and Co is infiltrated into an alumina carrier of which the primary particle has an aspect ratio of 30 or less. At this time, an org. solvent wherein the sum of the hydrogen bonding term and polar term of a solubility parameter is 40% or more of the sum of the intermolecular term, polar term and hydrogen bonding term of the solubility parameter is added to the support soln. The surface tension of the support soln. is set to 40dyn/cm or less before the alumina carrier is impregnated. By this constitution, the deterioration of the strength of the catalyst carrier can be suppressed when the catalyst carrier is impregnated with the support soln. to support active metal on the catalyst carrier and active metal is inexpensively supported with the catalyst carrier.

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(71)出願人　000231109
株式会社ジャパンエナジー
東京都港区虎ノ門二丁目10番１号

(72)発明者　岩田　好喜
埼玉県戸田市新曽南三丁目17番35号　株式会社ジャパンエナジー内

(72)発明者　加藤　晃
埼玉県戸田市新曽南三丁目17番35号　株式会社ジャパンエナジー内

(72)発明者　中島　章
埼玉県戸田市新曽南三丁目17番35号　株式会社ジャパンエナジー内

(74)代理人　弁理士　並川　啓志

(54)【発明の名称】　触媒担体の含浸方法

(57)【要約】

【構成】比較的脆い触媒への活性金属種の含浸の際発生する亀裂の抑制、機械的強度の低下防止、欠け、粉化を抑制するために、その担持液に溶解度パラメーターの分子間力項、極性項、水素結合項のうち、水素結合項と極性項の和が溶解度パラメーター全体の40%以上である有機溶剤を添加しその表面張力を40dyn/cm以下とした液を用いる。

【効果】本発明により、触媒が有する細孔内部の気体と担持液の気液置換を速やかに行わせることで、細孔内部の圧力上昇を抑制することができ、触媒表面や内部に発生する亀裂の抑制、機械的強度の改善、欠け、粉化の抑制等ができる。

1

【特許請求の範囲】

【請求項１】　一次粒子のアスペクト比が30以下であるアルミナ担体にMo,Ni及びCoからなる活性金属を単独若しくは２種以上を含む担持液を含浸する方法において、前記担持液に溶解度パラメーターの水素結合項と極性項の和が溶解度パラメーターの分子間力項、極性項、水素結合項の和の40%以上である有機溶剤を添加して、該担持液の表面張力を40dyn/cm以下としてから含浸することを特徴とする触媒担体の含浸方法。

【発明の詳細な説明】

【０００１】

【産業上の利用分野】本発明は、触媒担体に活性金属のMo、Ni、Co、Mo-Ni、Mo-Co等を担持する際に用いられる含浸方法に関するものであり、特に一次粒子のアスペクト比が30以下の粉体で形成される比較的脆い触媒担体の含浸時における強度劣化を抑制する方法である。

【０００２】

【従来技術】触媒担体は、粉体を原料として一般的にこの粉体を混練、押し出し、焼成して作製される。触媒担体はその触媒活性を上げるため細孔径が数十ｎｍ以下と小さく、その細孔径分布がシャープであることが必要である。このような触媒担体の細孔内に活性金属を担持するには、触媒担体を噴霧含浸、浸漬含浸などの含浸の後、乾燥焼成を行う。噴霧含浸は、活性金属を含んだ担持水溶液を触媒担体に噴霧することにより触媒担体に活性金属を担持する方法であり、また浸漬含浸は、触媒担体を活性金属を含む担持水溶液に浸漬することで触媒担体に活性金属を担持する方法である。ところが、含浸の際、速やかに触媒が有する細孔内部の気体と担持液の気液置換が行われないために生ずる圧力上昇によって触媒担体に亀裂、欠け、粉化が発生したり、機械的強度が更に低下するという問題点がある。

【０００３】ｷｬﾀﾘｽﾄ ｻﾎﾟｰﾂ ｱﾝﾄﾞ ｻﾎﾟｰﾃｨﾄﾞ ｷｬﾀﾘｽﾂ(Catalyst Supports And Supported Catalysts;P40-41(1987))には、アルミナ担体に担持液を含浸する際、触媒担体の細孔内の担持液の気泡の圧力Ｐはヤングーラプラス式則に従う次式で示されている。

$$P - P_0 = (2\gamma / r)\cos\alpha$$

ここで、P_0；大気圧、γ；気固界面における表面張力、ｒ；触媒担体の細孔半径、α；担持液の担体への接触角である。アルミナー水の場合、$\cos\alpha = 1$、$\gamma = 7 \times 10^2 N \cdot m^{-1}$を例にとり、細孔半径が２．５nmであると、細孔内の気泡に生じる圧力は、５６０barと非常に高くなる。この圧力が含浸の際、触媒を破壊する原因であることが示されている。そして、その解決策としてこの気泡が、水に溶解、拡散し、触媒担体外に拡散しやすいように減圧下で含浸を行う脱気含浸が有効であると述べている。

【０００４】

【発明が解決しようとする課題】しかし、脱気含浸は、

2

設備のコストが高いという問題点がある。更に、一次粒子のアスペクト比が３０以下の触媒担体は、細孔径分布がシャープであるため良好な触媒活性が得られるが触媒担体強度としては比較的脆く、従来の担持水溶液では細孔径の分布が小さいため速やかに細孔内部の気体との気液置換が行われず含浸時の強度劣化が特に大きいという問題点がある。本発明は、上記の問題点に鑑み、担持液を触媒担体に含浸させ、活性金属を担持する際の触媒担体の強度劣化を抑制することができる安価な触媒担体の担持方法を提供するものである。アスペクト比とは粉体の一次粒子の短軸と長軸の比のことを言い、例えば粉体の一次粒子の透過型電子顕微鏡によって写真を撮り、無差別に10個の粒子について短軸、長軸の長さをはかり、その比を求めたものである。

【０００５】

【課題を解決するための手段】本発明者らは上記問題点を解決するため、担持液、特にその表面張力に着目して鋭意検討を続けた結果、担持液の表面張力を有機溶剤の添加で制御することにより、触媒の細孔内部の気体と担持液の気液置換が速やかに行われないために生ずる圧力上昇によって発生する触媒表面や内部の亀裂の抑制、機械的強度の改善、粉化の抑制等ができることを見い出した。

【０００６】すなわち、本発明は、一次粒子のアスペクト比が30以下であるアルミナ担体にMo,Ni,Coからなる活性金属を単独若しくは２種以上を含む担持液を含浸する方法において、前記担持液に溶解度パラメーターの水素結合項と極性項の和が溶解度パラメーターの分子間力項、極性項、水素結合項の和の40%以上である有機溶剤を添加して、該担持液の表面張力を40dyn/cm以下としてから含浸することを特徴とする触媒担体の含浸方法である。

【０００７】本発明の理解を容易にするため、具体的かつ詳細に説明する。触媒担体の基本組成は主として、Al_2O_3であり、数十nm以下の細孔を有している。そして、その一次粒子のアスペクト比は30以下のものである。担持液は、活性金属であるMo、Ni、Coを含む塩を単独また複数溶解した液である。例えば、一般的に用いられる塩は、ヘプタモリブデン酸アンモニウム塩、硝酸コバルト塩、硝酸ニッケル塩、炭酸コバルト塩、炭酸ニッケル塩等である。

【０００８】本発明者らは、上記の粉末で作製された担体に様々な表面張力を有する担持液を含浸し、機械的強度や亀裂の発生状況を比較した。その結果、担持液の表面張力が40dyn/cmより大きいと、触媒が有する細孔内部の気体と担持液との気液置換が速やかに行われず、細孔内部の圧力上昇が発生し、触媒表面に亀裂が生じ機械的強度が低下する。従って、担持液の表面張力は40dyn/cm以下にすることが有効である。

【０００９】担持液の表面張力を40dyn/cm以下に下げる

には、担持液と相溶性のある有機溶剤を添加する方法と界面活性剤を添加する方法がある。担持液に界面活性剤を添加して表面張力を低下させることを試みた場合、このような界面活性物質はそのほとんどが気液界面に集まり、見かけ上、担持液の表面張力は減少するものの実際に表面張力が減少しているのは担持液の表面近傍の極一部に限られ、その他の大部分の溶液部には界面活性剤の寄与がないので表面張力が減少しない。このため、このような界面活性剤を含んだ担持液で噴霧含浸を行うと、結果として高い表面張力をもつ液滴が含浸され、亀裂の発生、機械的強度の低下、欠け、粉化等が生じる。このことから担持液の表面張力の低下は、あくまで個々の溶剤分子間の凝集力の低下により得られることが必要であり、界面活性剤の使用は好ましくない。

【００１０】有機溶剤の溶解度パラメーターの水素結合項と極性項の和が溶解度パラメーターの分子間力項、極性項、水素結合項の和の40%以上である有機溶剤は水と一定の相溶性を有し、水より表面張力が低い。従って、このような有機溶剤を添加すると水溶液の表面張力を下げることができる。一般概念として、界面活性剤と有機溶剤とでは一部にオーバーラップする範疇の液体であり、厳格な区別が困難な場合があるが、本発明の範囲で規定する有機溶剤では界面活性剤的性質、すなわち、気体-液体界面に自発的に集まるような性質を有するものは含まない。本発明者らは上記の有機溶剤のうち一種もしくは複数を担持水溶液を添加して表面張力を40dyn/cm以下にした担持液を作製し試験を行った。その結果、含浸後の担体の機械的強度が改善され触媒の含浸工程が好適に実施できることがわかった。本発明で使用可能な有機溶剤は、溶解度パラメーターの水素結合項と極性項の和が溶解度パラメーターの分子間力項、極性項、水素結合項の和の40%以上であり、具体的にはアルコール類、ケトン類、カルボン酸類、エーテル類、エステル類等があるが、効果の程度、入手の容易さ、安全性、コスト等から、エタノール、プロパノール、ブタノール、アセトン、メチルエチルケトン等がより好ましい。これらは触媒の活性に影響を与えない。溶解度パラメーターの水素結合項と極性項の和が溶解度パラメーターの分子間力項、極性項、水素結合項の和の40%未満である有機溶剤は、水との相溶性が乏しいため担持液の表面張力を40dyn/cm以下まで低下させる量を添加すると相分離して好適に実施できない。

【００１１】尚、溶解度パラメーター（δ）とは、物質の凝集エネルギー密度の平方根であり、$\delta=((\delta d)^2+(\delta p)^2+(\delta h)^2)^{1/2}$で表される。なお、δdは分子間力項、δpは極性項、δhは水素結合項である。本発明で使用できる有機溶剤は、$(\delta p+\delta h)/(\delta d+\delta p+\delta h)\geqq 0.40$である有機溶剤である。以下本発明の実施例について説明する。

【００１２】

【実施例１】平均アスペクト比30以下の一次粒子から形成され、10nm程度の中心細孔径を有し、担体強度11.3MPa、亀裂を有する担体の割合20%の触媒担体（平均触媒径；1/12インチ）にヘプタモリブデン酸アンモニウム塩水溶液に溶解度パラメーターの分子間力項、極性項、水素結合項の和のうち極性項と水素結合項の和の割合が57%であるイソプロパノールを濃度5mol%混在させた担持液（表面張力35dyn/cm）を噴霧含浸し、130℃で乾燥した。乾燥後の強度は、10.9MPaに、亀裂の有する割合は40%、粉化した割合は5%であった。これを更に、同等なイソプロパノールを濃度5mol%を含む表面張力35dyn/cmを有するニッケルの硝酸塩の担持液を連続的に同様に含浸すると強度は10.8MPaに、亀裂の有する割合は45%、粉化した割合は10%となった。このように、含浸による触媒担体の強度劣化は小さく、また、亀裂の有する割合、粉化する触媒の割合の増加も少ない。これを500℃で焼成し得られた触媒の脱硫活性、脱金属活性は比較例の触媒と同等であった。

【００１３】

【比較例１】10nmの中心細孔径を有していて、担体強度11.3MPa、亀裂を有する割合20%の触媒担体にモリブデンの担持量8～10wt%を溶解したヘプタモリブデン酸アンモニウム塩水溶液の担持液（表面張力70dyn/cm）を噴霧含浸し130℃で乾燥した。その強度は6.1MPaに、亀裂の有する割合は80%、粉化する割合は50%となった。これを更に、表面張力69dyn/cmを有するニッケルの硝酸塩の担持液を連続的に同様に含浸、乾燥を行うと強度は5.8MPaに低下し、亀裂の有する割合は90%、粉化する割合は60%に増加した。

【００１４】

【比較例２】実施例１の担持液のイソプロパノールの代わりにノニオン系の界面活性剤であるポリオキシエチレンアルキルエーテルを0.1wt%混在させたモリブデン系担持液（表面張力30dyn/cm）及びニッケル系担持液（表面張力35dyn/cm）を用い実施例１と同様に噴霧含浸し130℃で乾燥した。乾燥後の強度は、7.4MPaに、亀裂の有する割合は73%、粉化する割合は、40%であった。また、同様の界面活性剤を含むニッケル系の担持液で含浸、乾燥を行うと、強度は7.1MPaに、亀裂の有する担体の割合は80%、粉化した担体の割合は40%となった。比較例１と同様に、触媒担体の強度劣化が大きく、また、亀裂の有する割合、粉化する触媒の割合の増加も大きい。

【００１５】実施例１のイソプロパノールを担持液に混在させると溶液全体に分散し、溶液全体の表面張力が減少するのに対して、比較例２の界面活性剤のポリオキシエチレンアルキルエーテルでは活性種のほとんどが気液界面に集まり、気液界面の表面張力は減少するもののその他の大多数の溶液部は表面張力が減少しないので噴霧含浸を行うと高い表面張力を持つ液滴が含浸され、亀裂

の発生、機械的強度の低下、欠け、粉化の発生が生じる。

【００１６】

【発明の効果】以上説明したように、溶解度パラメーターの極性項と水素結合項の和が溶解度パラメーターの分子間力項、極性項、水素結合項の和の40%以上である有機溶剤から選ばれる一種、若しくは複数を含むことで表面張力を40dyn/cm以下とした担持液に含浸することで、触媒が有する細孔内部の気体と担持液の気液置換を速やかに行わせ、生ずる圧力上昇を抑制することができ、触媒表面や内部に発生する亀裂で40%程の抑制、機械的強度で1.5倍程の改善、欠け、粉化で50%程の抑制ができるようになった。また、実施例１、比較例１（従来法）で作製された触媒の活性試験（脱硫活性、脱金属活性）を行

ったところ、その活性に差はなかった。特公平2-35710の耐火物の製造法において耐火物の成形物にコロイド状シリカ液と界面活性剤または水溶性有機物とからなる担持液を含浸させ、必要に応じて乾燥または焼成する事および、この方法により耐火物の圧縮破壊強度が向上できることが示されているが、対象が耐火物であり、また液がコロイド状シリカ液と本方法の液である触媒担持液とは異なる。また、特公昭62-42743のアルキレンオキシサイド製造用触媒では、アルカリ金属含浸液の溶媒に誘電定数8以下の溶剤を使用することで、銀担持触媒にアルカリ金属を特定的に導入することを可能とするものであるが、本方法の目的は機械的強度の低下の抑制であり目的が全く異なる。

フロントページの続き

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* NOTICES *

1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Industrial Application] This invention is the approach of controlling degradation on the strength at the time of sinking [of the comparatively weak catalyst support in which especially the aspect ratio of a primary particle is formed by 30 or less fine particles] in about the sinking-in approach used in case Mo, nickel and Co of an active metal, Mo-nickel, Mo-Co, etc. are supported to catalyst support.
[0002]
[Description of the Prior Art] Generally, it was kneading and extruding, catalyst support is calcinating fine particles, and these fine particles are produced considering them as a raw material. The pole diameter of catalyst support is as small as dozens of nm or less in order to raise the catalytic activity, and it needs for the pore volume distribution to be sharp. In order to support an active metal in the pore of such catalyst support, desiccation baking is performed after sinking [catalyst support] in, such as spraying sinking in and immersion sinking in. Spraying sinking in is the approach of supporting an active metal to catalyst support by spraying the support water solution containing an active metal on catalyst support, and immersion sinking in is the approach of supporting an active metal with catalyst support being immersed in the support water solution containing an active metal to catalyst support. However, there is a trouble that a crack, a chip, and powdering occur in catalyst support by the pressure buildup produced since the vapor-liquid permutation of the gas inside the pore which a catalyst has promptly, and support liquid is not performed, or a mechanical strength falls further, in the case of sinking in.
[0003] catalyst SAPOTSU and -- SAPOTIDO In case support liquid is sunk into alumina support, the pressure P of the air bubbles of the support liquid in the pore of catalyst support is shown to KYATARISUTSU (Catalyst Supports And Supported Catalysts;P 40-41 (1987).) by the degree type according to a young-Laplace style rule.
P-P0=(2 gamma/r) cosalpha -- here, it is a contact angle to P0; atmospheric pressure, the surface tension in gamma; gas-solid interface, the pore radius of r; catalyst support, and the support of alpha; support liquid. In the case of alumina-water, the pressure produced to the air bubbles in pore as cosalpha=1 and gamma=7x102 N-m -1 are taken for an example and a pore radius is 2.5nm becomes very high with 560bar. In case this pressure is sinking in, it is shown that it is the cause which destroys a catalyst. And it is said that degassing sinking [which sinks in under reduced pressure so that these air bubbles may dissolve in water, and may diffuse and it may be easy to diffuse them out of catalyst support as that solution] in is effective.
[0004]
[Problem(s) to be Solved by the Invention] However, degassing sinking in has the trouble that the cost of a facility is high. Furthermore, since 30 or less catalyst support has sharp pore volume distribution, although good catalytic activity is acquired in the aspect ratio of a primary particle, as catalyst support reinforcement, it is comparatively weak, and in the conventional support water solution, since distribution of a pole diameter is small, a vapor-liquid permutation with the gas inside pore is not performed promptly, but there is a trouble that degradation on the strength at the time of sinking in is especially large. In view of the above-mentioned trouble, this invention infiltrates support liquid into catalyst support, and offers the support approach of the cheap catalyst support which can control degradation of the catalyst support at the time of supporting an active metal on the strength. An aspect ratio means the ratio of the minor axis and major axis of the primary particle of fine particles, for example, a photograph is taken with the transmission electron microscope of the primary particle of fine particles, and the die length of a minor axis and a major axis is indiscriminately found for a scale and its ratio about ten particles.
[0005]

[Means for Solving the Problem] It found out that control of the crack of the catalyst front face generated by the pressure buildup produced since the vapor-liquid permutation of the gas inside the pore of a catalyst and support liquid is not performed promptly, or the interior, improvement of a mechanical strength, control of powdering, etc. could be performed by controlling the surface tension of support liquid by addition of an organic solvent, as a result of continuing examination wholeheartedly especially paying attention to the surface tension, support liquid and in order that this invention persons may solve the above-mentioned trouble.

[0006] Namely, this invention sets the active metal with which the aspect ratio of a primary particle becomes the alumina support which is 30 or less from Mo, nickel, and Co to independent or the approach of sinking in the support liquid containing two or more sorts. The organic solvent whose sum of the hydrogen bond term of a solubility parameter and a polar term is 40% or more of the sum of the intermolecular force term of a solubility parameter, a polar term, and a hydrogen bond term is added in said support liquid. It is the sinking-in approach of the catalyst support characterized by sinking in after making surface tension of this support liquid into 40 or less dyn/cm.

[0007] In order to make an understanding of this invention easy, it explains to concrete and a detail. The basic presentation of catalyst support is aluminum 2O3, and mainly has pore dozens of nm or less. And the aspect ratio of the primary particle is 30 or less thing. Support liquid is independent and liquid dissolved [two or more] about the salt containing Mo, nickel, and Co which are an active metal. For example, the salts generally used are a hepta-ammonium-molybdate salt, a cobalt nitrate salt, a nickel nitrate salt, a cobalt carbonate salt, a nickel carbonate salt, etc.

[0008] this invention persons sank the support liquid which has various surface tension into the support produced with the above-mentioned powder, and compared the mechanical strength and the crack initiation situation with it. Consequently, if the surface tension of support liquid is larger than 40 dyn/cm, the vapor-liquid permutation of the gas inside the pore which a catalyst has, and support liquid is not performed promptly, but the pressure buildup inside pore occurs, a crack will arise on a catalyst front face and a mechanical strength will fall to it. Therefore, as for the surface tension of support liquid, it is effective to make it 40 or less dyn/cm.

[0009] In order to lower the surface tension of support liquid to 40 or less dyn/cm, there are support liquid, an approach of adding an organic solvent with compatibility, and the approach of adding a surfactant. When to add a surfactant in support liquid and to reduce surface tension is tried, as for such a surface active substance, the most is restricted to a gas-liquid interface, seemingly, although the surface tension of support liquid decreases, it is restricted to the pole near the front face of support liquid part the assembly and that surface tension is actually decreasing, and since there is no contribution of a surfactant in the other [the great portion of] solution sections, surface tension does not decrease. For this reason, if the support liquid containing such a surfactant performs spraying sinking in, a drop with surface tension high as a result will sink in, and the fall of crack initiation and a mechanical strength, a chip, powdering, etc. will arise. It is required for the fall of the surface tension of support liquid to be obtained from this by the fall of the cohesive force between each solvent molecules to the last, and use of a surfactant is not desirable.

[0010] The organic solvent whose sum of the hydrogen bond term of the solubility parameter of an organic solvent and a polar term is 40% or more of the sum of the intermolecular force term of a solubility parameter, a polar term, and a hydrogen bond term has water and fixed compatibility, and its surface tension is lower than water. Therefore, if such an organic solvent is added, the surface tension of a water solution can be lowered. Although it may be the liquid of the criteria which overlap a part by the surface active agent and the organic solvent as a general concept and a strict distinction may be difficult, by the organic solvent specified in the range of this invention, what has a surface-active-agent-property, i.e., the property in which it gathers for a gas-liquid interface spontaneously, does not contain. this invention persons examined by producing the support liquid which added the support water solution for a kind or plurality among the above-mentioned organic solvents, and made surface tension 40 or less dyn/cm. Consequently, it turned out that the mechanical strength of the support after sinking in is improved and the sinking-in process of a catalyst can carry out suitably. Although the sum of the hydrogen bond term of a solubility parameter and a polar term is 40% or more of the sum of the intermolecular force term of a solubility parameter, a polar term, and a hydrogen bond term in this invention and an usable organic solvent specifically has alcohols, ketones, carboxylic acids, ether, and ester, the ethanol from extent of effectiveness, the ease of acquisition, safety, cost, etc., propanol, a butanol, an acetone, a methyl ethyl ketone, etc. are more desirable. These do not affect the activity of a catalyst. Since compatibility with water is scarce, if the amount in which the surface tension of support liquid is reduced to 40 or less dyn/cm is added, phase separation of the organic

solvent whose sum of the hydrogen bond term of a solubility parameter and a polar term is less than 40% of the sum of the intermolecular force term of a solubility parameter, a polar term, and a hydrogen bond term is carried out, and it cannot be carried out suitably.

[0011] In addition, a solubility parameter (delta) is the square root of the cohesive energy density of the matter, and it is expressed with delta=(deltad) (2+(deltap) 2+(deltah) 2) 1/2. In addition, deltad is [a polar term and deltah of an intermolecular force term and deltap] hydrogen bond terms. The organic solvent which can be used by this invention is an organic solvent which is (deltap+deltah)/(deltad+deltap+deltah) >=0.40. The example of this invention is explained below.

[0012]

[Example 1] It is formed from a 30 or less average aspect ratio primary particle, and has the main pole diameter of about 10nm. In a hepta-ammonium-molybdate salt water solution at the catalyst support (the diameter of an average catalyst; 1/12 inch) of 20% of rates of support which has support on-the-strength 11.3MPa and a crack The intermolecular force term of a solubility parameter, the isopropanol whose percentage of the sum of a polar term and a hydrogen bond term is 57% among the sums of a polar term and a hydrogen bond term -- concentration % of five mols -- it carried out spraying sinking in of the support liquid (surface tension 35 dyn/cm) made intermingled, and dried at 130 degrees C. The percentage which powdered the rate that a crack has the reinforcement after desiccation in 10.9MPa, 40% was 5%. The rate in which the rate that a crack has reinforcement in 10.8MPa(s) when it sinks in similarly continuously powdered the support liquid of the nitrate of the nickel which has surface tension 35 dyn/cm which contains [this] concentration % of five mols for still more equivalent isopropanol 45% became 10%. Thus, degradation of the catalyst support by sinking in on the strength is small, and there are also few increments in the rate which a crack has, and the rate of the catalyst to powder. The desulfurization activity of the catalyst which might be calcinated at 500 degrees C in this, and demetallization activity were equivalent to the catalyst of the example of a comparison.

[0013]

[The example 1 of a comparison] It carried out spraying sinking in of the support liquid (surface tension 70 dyn/cm) of the hepta-ammonium-molybdate salt water solution which dissolved amount [of molybdenum] 8 of support - 10wt% in the catalyst support of 20% of rates which has the main pole diameter of 10nm and has support on-the-strength 11.3MPa and a crack, and dried at 130 degrees C. The rate which powders the rate that a crack has the reinforcement in 6.1MPa(s), 80% became 50%. When sinking in and desiccation were performed similarly continuously, reinforcement fell to 5.8MPa(s), and the rate which the rate which a crack has powders 90% increased the support liquid of the nitrate of the nickel which has surface tension 69 dyn/cm for this further to 60%.

[0014]

[The example 2 of a comparison] the polyoxyethylene alkyl ether which is the surface active agent of the Nonion system instead of the isopropanol of the support liquid of an example 1 -- 0.1wt(s)% -- it carried out spraying sinking in as well as an example 1 using the molybdenum system support liquid (surface tension 30 dyn/cm) and nickel system support liquid (surface tension 35 dyn/cm) which were made intermingled, and dried at 130 degrees C. The percentage which powders the rate that a crack has the reinforcement after desiccation in 7.4MPa, 73% was 40%. Moreover, when the support liquid of the nickel system containing the same surfactant performed sinking in and desiccation, the rate of the support which powdered the rate of support that a crack has reinforcement in 7.1MPa(s), 80% became 40%. Like the example 1 of a comparison, degradation of catalyst support on the strength is large, and the increment in the rate which a crack has, and the rate of the catalyst to powder is also large.

[0015] As opposed to distributing in the whole solution, if the isopropanol of an example 1 is made intermingled in support liquid, and the surface tension of the whole solution decreasing In the polyoxyethylene alkyl ether of the surface active agent of the example 2 of a comparison, most active species to a gas-liquid interface An assembly, Although the surface tension of a gas-liquid interface decreases, since surface tension does not decrease, if it performs spraying sinking in, a drop with high surface tension will sink in and generating of the fall of crack initiation and a mechanical strength, a chip, and powdering will produce the solution section of other large majorities.

[0016]

[Effect of the Invention] As explained above, the sum of the polar term of a solubility parameter and a hydrogen bond term The intermolecular force term of a solubility parameter, By sinking into the support liquid which made surface tension 40 or less dyn/cm by a kind chosen from the organic solvent which is 40% or more of the sum of a polar term and a hydrogen bond term, or plurality being included The vapor-

liquid permutation of the gas inside the pore which a catalyst has, and support liquid could be made to be able to perform promptly, the pressure buildup to produce could be controlled, and about 50% of control was completed in about 40% of control with the crack generated in a catalyst front face or the interior, and came complete by the improvement of about 1.5 times, the chip, and powdering in a mechanical strength. Moreover, when the activity trial (desulfurization activity, demetallization activity) of the catalyst produced in the example 1 and the example 1 (conventional method) of a comparison was performed, there was no difference in the activity. Although it is shown infiltrating the support liquid which becomes the moldings of refractories from colloid silica liquid, a surfactant, or the water-soluble organic substance in the manufacturing method of the refractories of JP,2-35710,B, and drying or calcinating if needed, and that the compression disruptive strength of refractories can improve by this approach, it differs from the catalyst support liquid whose objects are refractories and whose liquid is colloid silica liquid and the liquid of this approach. Moreover, with the catalyst for alkyleneoxy side manufacture of JP,62-42743,B, although it makes it possible to introduce alkali metal into a silver support catalyst specifically by using an eight or less-dielectric constant solvent for the solvent of alkali-metal sinking-in liquid, the purpose of this approach is control of a fall of a mechanical strength, and the purposes completely differ.

[Translation done.]

* NOTICES *

1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

CLAIMS

[Claim(s)]
[Claim 1] The sinking-in approach of the catalyst support characterized for the active metal with which the aspect ratio of a primary particle becomes the alumina support which is 30 or less from Mo, nickel, and Co by independent or sinking in after adding the organic solvent whose sum of the hydrogen bond term of a solubility parameter and a polar term is 40% or more of the sum of the intermolecular force term of a solubility parameter, a polar term, and a hydrogen bond term in said support liquid and making surface tension of this support liquid into 40 or less dyn/cm in the approach of sinking in support liquid containing two or more sorts.

[Translation done.]